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[Page 1 of 2]

Date Dec. 19, 2003

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PROVISIONAL PATENT APPLICATION

FOR

ACTIVE ELECTRONIC DEVICES WITH NANOWIRE
COMPOSITE COMPONENTS

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Active electronic devices with nanowire composite components.

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Prior art

1. Electronic devices, such as resistors, diodes and transistors with nanowires as the conducting channel have been fabricated before. For the purpose of this application, nanowires are defined as structures where at least one dimension is less than 100nm, and the term includes bulk nanowires, nanofibres, nanoribbons and also carbon and other nanotubes.

(X. Duan and C. Lieber *Adv. Mat* 12, 298 (2000))

Adrian Bachtold, Peter Hadley, Takeshi Nakanishi, and Cees Dekker "Logic Circuits with Carbon Nanotube Transistors", *Science* 294, 1317-1320 (2001).

Derycke, V.; Martel, R.; Appenzeller, J.; Avouris, Ph.; "Carbon Nanotube Inter- and Intramolecular Logic Gates," *Nano Lett.* 1, 453-456 (2001).

Ali Javey, Moonsub Shim, and Hongjie Dai, "Electrical properties and devices of large-diameter single-walled carbon nanotubes," *Appl. Phys. Lett.* 80, 1064-1066 (2002).

R. Martel, T. Schmidt, H. R. Shea, T. Hertel, and Ph. Avouris "Single- and multi-wall carbon nanotube field-effect transistors," *Appl. Phys. Lett.* 73, 2447-2449 (1998).

Paul L. McEuen, Marc Bockrath, David H. Cobden, Young-Gui Yoon, and Steven G. Louie, "Disorder, Pseudospins, and Backscattering in Carbon Nanotubes," *Phys. Rev. Lett.* 83, 5098-5101 (1999).

Sander J. Tans, Alwin R. M. Verschueren, Cees Dekker, "Room-temperature transistor based on a single carbon nanotube," *Nature* 393, 49-52 (1998).)

2. It has also been shown that devices with carbon nanotube networks can also operate as transistor devices. Networks of nanotubes have also been shown to support Field Effect Transistor (FET) operation. (Snow, E. S., Novak, J. P., Campbell, P. M. & Park, D. *Random networks of carbon nanotubes as an electronic material. Applied Physics Letters* 82, 2145-2147 (2003).

J-C Gabriel: *Large Scale production of Carbon Nanotube Transistors. Mat.Res. Soc. Symp. Proc. Vol 776*

R.J.Chen et al: *Noncovalent functionalization of carbon nanotubes for highly specific biosensors PNAS* 100, 49483 (2003)

K. Bradley, J-C P Gabriel and G. Gruner: *Flexible Nanotube Electronics Nano Lett* 2003
N.P.Armitage, J-C P Gabriel and G.Gruner *Langmuir-Blodgett nanotube films Appl. Phys. Lett* submitted 2003)

3. Recently several groups have also fabricated composites with nanowires as one of the components and have been demonstrated that the composites can be used as resistors. (Chen, R.Ramasuramanian and H.Liu *Noncovalent engineering of Carbon nanotube Surfaces*

P. Ramamurthy et al ; *Polyaniline/Single walled carbon nanotube Composite Electronic Device 11th Foresight Conference*)

The Innovation

4. This invention proposes an active electronic device structure where a composite material that incorporates nanowires (as defined above) acts as element of the device. A composite is a material that consists of a homogenous, or nearly homogenous (or uniform) mixture two or several components, with one component a nanowire (Fig.1). the nanowires are dispersed uniformly or nearly uniformly within the "host material" that can be a polymer or any other material in which the nanowires can be embedded.

One example is the structure where the composite is the conducting channel that connects the source and the drain - of the structure. Nano-scale electronic devices such as diodes, field effect transistors, and logic elements are included in this application. A typical transistor device architecture is shown in Fig. 2, while in Fig. 3 a typical diode configuration is displayed. Logic elements can be fabricated by utilizing transistors with p and n-type characteristics.

Controlling the density of the nanowire density (see Fig.1) in the composite array is essential for appropriate device operation. For a dense array screening of the gate voltage by the wires is important, in a fashion similar to gate voltage screening due to a metal layer deposited on the device. For a rarified array such screening is not important and the array can serve as the source-to-drain conducting channel. For an array involving both metallic and semiconducting wires (such as in case of carbon nanotubes) the conductance of the off state - which can be reached by an application of a positive gate voltage - will be dominated by the conductance of the metallic tubes. It is expected that arrays close to, and on the conducting side of the two dimensional percolation limit will have appropriate transistor characteristics. Under such circumstances screening effects are expected to be small, but conduction is still provided by the nanowire network. Note that for even a poorly conducting substrate, a conducting channel can form where part of the current is supported not by the nanotube but by the substrate.

5. With appropriate doping and surface modification both n-type and p-type composite behavior can be fabricated. Fabricating n-type devices using polymers have been demonstrated before, and p-type transistor operation due to absorbed oxygen has also been shown. In case of semiconducting nanowires, bulk doping can lead to p or n-type operation. A combination and p and n-type transistor devices can lead to a logic element in a fashion similar to logic elements fabricated using standard transistor devices. (Star, A.; Bradley, K.; Gabriel, J.-C.; Gruner, G. "Nano-Electronic Sensors: Chemical Detection Using Carbon Nanotubes," *PMSE*, 2003, (in press)
Star, A.; Han, T.-R.; Joshi, V.; Gruner, G. "Polymer Coatings of Carbon Nanotube Sensors," *Polymer Prep.* 2003, 44(2), 201.
Star, A.; Gabriel, J.-C. P.; Bradley, K.; Gruner, G. "Electronic Detection of Specific Protein Binding Using Nanotube FET Devices," *Nano Lett.* 2003, 3, 459-463.)

6. The conducting and insulating elements of the devices can be fabricated of various appropriate materials, including metals, conducting and non-conducting polymers and also composites. In the latter case, the constituent materials of the composites, together

with the ratio of the constituents in the composite determines the conducting properties of the components of the device.

A variety of nanowire composites can be fabricated and incorporated in the device architecture. They include small gap oxide semiconducting wires, transition metal-chalcogen molecular nanowires, planar organic molecule based wires, polymeric nanofibres and various single and multiwall nanotube structures (*Nanoelectronic Devices Based on Nanowire Networks UCLA provisional application 2003 R.Kaner, G.Gruner and J.Huang inventors*).

Various composite fabrication routes are available, and all can be applied to fabricate composites with the appropriate electrical and other properties. A partial list of fabrication methods is listed as Appendix.

Captions

1a. A field effect transistor device with a nanowire composite conducting channel. The substrate and the gate can be made of metallic, or polymeric materials. A typical bottom ate configuration is shown, but other transistor configurations known in the literature can also be fabricated. The source, (S), drain (D) and the gate can be made of various conducting materials, including nanowire composites of various composition, and density.

1b. Top view of the nanowire composite with the full lines indicating the nanowires.

Appendix: Prior art fabrication routes of nanowire composites

1. Homogeneous carbon nanotube/polymer composites for electrical applications

Rajagopal Ramasubramaniam, Jian Chen, and Haiying Liu

Applied Physics Letters Vol 83(14) pp. 2928-2930. October 6, 2003

Abstract

Homogeneous carbon nanotube/polymer composites were fabricated using noncovalently functionalized, soluble single-walled carbon nanotubes (SWNTs). These composites showed dramatic improvements in the electrical conductivity with very low percolation threshold (0.05–0.1 wt % of SWNT loading). By significantly improving the dispersion of SWNTs in commercial polymers, we show that only very low SWNT loading is needed to achieve the conductivity levels required for various electrical applications without compromising the host polymer's other preferred physical properties and processability. In contrast to previous techniques, our method is applicable to various host polymers and does not require lengthy sonication. ©2003 American Institute of Physics.

2. Deformation of carbon nanotubes in nanotube–polymer composites

C. Bower, R. Rosen, L. Jin, J. Han, and O. Zhou

Applied Physics Letters Vol 74(22) pp. 3317-3319. May 31, 1999

Abstract

Composites of uniaxially oriented multiwalled carbon nanotubes embedded in polymer matrices were fabricated and investigated by transmission electron microscopy. In strained composite films, buckling was ubiquitously observed in bent nanotubes with large curvatures. By analyses of a large number of bent nanotubes, the onset buckling strain and fracture strain were estimated to be $\approx 5\%$ and $\approx 18\%$, respectively. The buckling wavelengths are proportional to the dimensions of the nanotubes. Examination of the fracture surface showed adherence of the polymer to the nanotubes. ©1999 American Institute of Physics.

3. Session W26 - Focus Session: Carbon Nanotube Composites.

FOCUS session, Thursday morning, March 06

Room 17B, Austin Convention Center

[W26.001] Polymer-grafted single-walled carbon nanotube composites

Gunaranjan Viswanathan (Dept. of Chemical Engineering, Rensselaer Polytechnic Institute, Troy, NY), Nirupama Chakrapani, Pulickel M. Ajayan (Dept. of Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, NY), Chang Y. Ryu (Dept. of Chemistry, Rensselaer Polytechnic Institute, Troy, NY)

Carbon nanotube-polymer composites hold great promise but the dispersion of nanotubes and the interface between the nanotubes and the polymer matrix are issues critical to successful applications. Chemical functionalization of the nanotube surface is a feasible approach towards solving these problems. But the methods employed tend to alter the original structure of the nanotubes. We have developed a novel route for grafting polystyrene chains onto pristine single-walled carbon nanotubes through a single-step anionic polymerization scheme, with the aim of improving the interface in these composites. The thermal properties of the composites are characterized using Differential Scanning Calorimetry and Thermogravimetric Analysis. The efficacy of the grafting mechanism is also evaluated.

[W26.002] SWNT/Polymer Composites Using Water Soluble Polymers

Xiefei Zhang, Tao Liu, Satish Kumar (School of Textile and Fiber Engineering, Georgia Institute of Technology, Atlanta, GA 30332), Vallerie Moore, R.H. Hauge, R.E. Smalley (Center for Nanoscale Science and Technology, Rice University, Houston, TX 77005)

Poly (vinyl pyrrolidone) (PVP) wrapped single wall carbon nano tubes (SWNTs) have been dispersed in water using surfactants. These dispersions have been used to process polymer/SWNT composite films utilizing water soluble polymers such as poly (vinyl alcohol) (PVA) and poly (acrylic acid) (PAA) as the matrix systems. Structure, morphology, and mechanical properties of these films have been studied. The infra-red absorption band of the C=O stretching vibration in PAA/SWNT shifted to lower wavenumber when compared to the pure polymer film. These and other property changes in PAA and PVA have been explained arising from the charge transfer between the polymer molecules and SWNTs. Dynamic mechanical transitions in PAA and PVA have also been effected with the presence of SWNTs.

[W26.003] Molecular Design of Strong SWNT/Polyelectrolyte Multilayers Composites

Nicholas Kotov (Oklahoma State University)

The mechanical failure of hybrid materials made from polymers and single wall carbon nanotubes (SWNT) is primarily attributed to poor matrix-SWNT connectivity and severe phase segregation. Both problems can be successfully mitigated when the SWNT composite is made following the protocol of layer-by-layer assembly. This deposition technique prevents phase segregation of the polymer/SWNT binary system, and after subsequent cross-linking, the nm-scale-uniform composite with SWNT loading as high as 50 wt% membranes delaminated from the substrate were found to be exceptionally strong with tensile strength approaches that of hard ceramics. Considering the light-weight nature of SWNT composites the prepared free-standing membranes can serve as unique components for a variety of long-life-time devices. The assembly process also affords preparation of aligned SWNT composites. Laminar flow adsorption shows gradual untangling of the nanotube agglomerates and the formation of parallel ribbons.

[W26.004] Single-Walled Carbon Nanotube/PMMA Composites

Fangming Du, John Fisher, Karen Winey (University of Pennsylvania, Dept. of Chemical Engineering, Dept. of Materials Science and Engineering, Philadelphia, PA)

Single-walled carbon nanotubes (SWNTs) have demonstrated unique mechanical, thermal and electrical properties. Similar properties are expected for polymer/SWNT nanocomposites. A new processing method has been used to produce PMMA/SWNT composites, which provides better dispersion of SWNT in the polymer matrix. Optical microscopy of the samples show improved dispersion of SWNT in the PMMA matrix, which is the key factor of the composite performance. Aligned and unaligned composite samples have been made for both purified SWNT and functionalized SWNT with different SWNT loadings. The tensile, thermal conductivity, and electroconductivity measurements of these samples will be performed.

[W26.005] Dispersion and Mechanical Properties of Carbon Nanotube/Polymer Composites via Melt Compounding

Russell Gorga, Robert Cohen (Massachusetts Institute of Technology)

This work is focused on the fabrication of carbon nanotube/polymer composites via melt compounding. The main objective of this work is to realize the outstanding properties of carbon nanotubes (high modulus, high thermal and electrical conductivity, elastic buckling) at the macroscopic level by blending carbon nanotubes into a polymer matrix. The challenge lies in dispersing these one dimensional nanoparticles in the polymer matrix. Dispersion of the nanotubes in the composites is analyzed via transmission and scanning electron microscopy. Mechanical properties as well as electrical and thermal conductivity are measured as a function of nanotube loading, orientation, and extrusion conditions. Multi-wall nanotube loadings in the range of 1 and 10 wt% concave-downward departures from the linear stress-strain behavior of the unmodified polymer below 50 observations are discussed in the context of possible deformation mechanisms for the nanotube composites.

[W26.006] Alignment of SWNT in Electrospun SWNT-Polymer Composite Fibers

I.N. Ivanov, A.A. Puztzy, M.J. Lance, S. Jesse, S. Viswanathan, P.F. Britt, D.B. Geohegan, Oak Ridge National Laboratory Collaboration

Alignment of single-wall carbon nanotubes within polymer composites is critical for applications requiring anisotropic electrical and thermal conductivity. Stable solutions of SWNT and polymers (including thermosets and biocompatible polymers such as PMMA, PEO, PVA, PVAc, PC) were electrospun to produce composite fibers with diameters varying from a few nanometers to hundreds of microns. High degrees of alignment of SWNT along the axis of the fibers were achieved as characterized by polarized microRaman spectroscopy of the SWNT within the fiber (up to 18:1 intensity ratio) for fibers of diameter less than ~ 20 microns. The strain-induced shift of the 2600 cm⁻¹ Raman band of the SWNT inside the composites was used to investigate the load transfer to the nanotubes during their controlled mechanical loading. The ability to produce highly-aligned SWNT within electrospun polymer composite fibers offers numerous opportunities for nanotube-based membranes, fabrics, and other structures which will be discussed.

[W26.007] Imaging and Electrical Characterization of Nanotube Networks in SWNT/Polymer Composites

Stephen Jesse (Dept. of Materials Science and Engineering, University of Tennessee), M.A. Guillorn, D.W. Austin, I.N. Ivanov, A.A. Ppuretky, S. Viswanathan, P.F. Britt, D.B. Geohegan, Oak Ridge National Laboratory Collaboration

In addition to enhanced mechanical properties, interconnected networks of carbon nanotubes are envisioned to provide directional thermal and electrical conductivity paths within multifunctional polymer composites. Their sensitivity to the surrounding polymer matrix and environment are envisioned as the basis for novel electronic devices as well as chemical, biological and photonic sensor arrays. Advancement in these areas requires fundamental understanding of the conduction within nanotube bundles and their junctions which form the composite network. To this end, we have investigated the current-voltage response of 100 nm - 800 nm thick SWNT/PMMA composite films using arrays of lithographically patterned electrodes of various geometries. SEM and AFM images of the composite/electrode configuration revealed the structure of the conductive network within the polymer matrix. Voltage contrast and EBIC (electron beam induced current) scanning electron microscopy are used to map paths of current flow through the composite. In addition, potential maps of charged composites are generated using atomic force microscopy. Image analysis of microscopy data was used to determine the number of connections, the distances between connections, and the connectivity of the network. Correlation of network geometry, current-voltage response, and node-voltage analysis was used to estimate the average resistivity of nanotube bundles and their interconnections. The nature and cause of non-ohmic responses measured in the composites of various nanotube weight loadings will be discussed.

[W26.008] SINGLE-WALLED CARBON NANOTUBE / NYLON66 COMPOSITES

Reto Hagenmueller, John E. Fischer, Karen I. Winey (Dept. of Materials Science and Engineering, University of Pennsylvania, Philadelphia, PA)

Previous work in our group has used a combination of solution and melt mixing to produce composites of either amorphous or semicrystalline thermoplastics and SWNT. Subsequently, these composites were subjected to melt fiber spinning that produces extraordinary alignment of the SWNT. The elastic moduli of SWNT/PMMA and SWNT/PE composites increase with both nanotube alignment and loading. The dispersion of the nanotubes in composites depends critically on the state of the nanotubes before the composite preparation. Nanotubes suspended in solvents are most promising. An interfacial in situ polymerization with either purified or functionalized nanotubes enables the fabrication of SWNT/nylon66 composites. Effects of the nanotubes on the crystallinity and melting temperatures are investigated. The mechanical, electrical, and thermal properties of isotropic films and/or anisotropic fibers of the SWNT/nylon66 composites are measured. The side groups of functionalized nanotubes are varied in terms of functionality and length to probe the interface in composites.

[W26.009] Carbon Nanotubes - Polymer Composites with Enhanced Conductivity using Functionalized Nanotubes

Rajagopal Ramasubramaniam, Jian Chen, Rishi Gupta (Zyvox Corporation, 1321 North Plano Road, Richardson, TX 75081)

Individual carbon nanotubes show superior electrical, mechanical and thermal properties [1]. Composite materials using carbon nanotubes as fillers are predicted to show similar superior properties. However, realization of such composites has been plagued by poor dispersion of carbon nanotubes in solvents and in polymer matrices. We have developed a method to homogeneously disperse carbon nanotubes in polymer matrices using functionalized nanotubes [2]. Thin films of functionalized single walled nanotubes (SWNT) - polystyrene composites and functionalized SWNT - polycarbonate composites were prepared using solution evaporation and spin coating. Both of the composites show several orders of magnitude increase in conductivity for less than 1 wt thresholds of the composites are less than 0.2 wt nanotubes. We attribute the enhanced conduction to the superior dispersion of the functionalized nanotubes in the polymer matrix and to the reduced nanotube waviness resulting from the rigid backbone of the conjugated polymer.

References: [1]. R. H. Baughman, A. A. Zakhidov and W. A. de Heer, *Science* v297, p787 (2002); [2]. J. Chen, H. Liu, W. A. Weimer, M. D. Halls, D. H. Waldeck and G. C. Walker, *J. Am. Chem. Soc.* v124, p9034 (2002).

[W26.010] Electrical and Dielectric Properties of Single Wall Carbon Nanotube Polymer Composites

Cheol Park, Kristopher E. Wise (ICASE), Zoubeida Ounaies (Virginia Commonwealth University), Sharon E. Lowther, Emilie J. Siochi, Joycelyn S. Harrison (Advanced Materials and Processing Branch, NASA Langley Research Center), Cosmas Chiteme, David S. McLachlan (Department of Physics, University of the Witwatersrand, Johannesburg, South Africa)

Single wall carbon nanotube (SWNT) reinforced polymer nanocomposites were prepared. Uniform dispersion of thin bundles of SWNTs in the polymer matrix was evidenced by high-resolution electron microscopy. Electrical and dielectric properties of the nanocomposites were measured as a function of SWNT concentration, temperature, and frequency. The nanocomposites exhibited capacitive behavior under the percolation threshold (0.05 volconductive behavior above. The relative dielectric constant increased significantly with increasing SWNT loading. Anomalous dielectric behavior of the nanocomposites was observed at high frequencies. The experimental results were fitted using a 2-parameter phenomenological percolation equation (PRB 56, 1236 (1998)). Analytical modeling and numerical simulation were carried out to aid in understanding the experimental results.

[W26.011] Dielectric properties of nanotube composites

Krzysztof Kempa (Boston College)

We investigate the dielectric response of composites made of short sections of multiwall nanotubes embedded in a dielectric medium such as polymer. We show that the resulting composite medium can have a wide range of dielectric properties, depending on the concentration of the nanotubes. The dielectric constant can be lower or higher than that of the surrounding dielectric medium. There is also a frequency range within which it becomes negative. We discuss the possible applications of such composites, including one suitable for subwavelength radio antennas.

[W26.012] Irradiation Effects on Multi-walled Nanotube-Polymer Composites

Julie P. Harmon, Shelli Tatro (Chemistry Department, University of South Florida), Apparao Rao (Physics Department, Clemson University)

Poly(methyl methacrylate) (PMMA) and Multi-walled nanotube (MWNT)/PMMA composites were irradiated in air. The constant dose rate was 985 rad/min at a dose of 5.7 Mrad using a Cesium-137 source. The samples were then analyzed by dielectric analysis (DEA), dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC), and Vickers microhardness. The MWNT composites were found to show increased radiation hardness with respect to the glass transition temperature and mechanical properties. Further, in initial aging studies for a period of three months composites show nearly complete recovery, while neat samples show increased degradation. Initial dielectric results show that electrical properties changed more significantly for the composites than for the neat PMMA.

[W26.013] Structure and Properties of Polymer Composites from Infiltrated Aligned Multiwall Carbon Nanotubes

I.N. Ivanov, A. Rar, A.A. Puretzky, M.J. Lance, S. Jesse, S. Viswanathan, P.F. Britt, G.P. Pharr, D.B. Geohegan, Oak Ridge National Laboratory Collaboration

Densely-packed, vertically-aligned multiwall carbon nanotubes (VA-MWNTs) can be grown to near millimeter thicknesses, offering the opportunity for thick, pre-aligned polymer composites with anisotropic thermal and electrical properties. Vertically-aligned arrays of MWNT were infiltrated with aminoepoxy and polymer solutions and cured. The resulting nanocomposite films were characterized by nanoindentation, SEM and optical microscopy, and microRaman spectroscopy. We found that the mechanical properties are strongly dependent on the nanotube orientation in the composites. For aminoepoxy, the height of the VA-MWNTs was preserved in the polymer composite. Enhancements in Young's modulus over pure epoxy were found in both parallel and perpendicular nanotube orientations, and ranged from 25(perpendicular) to 50composite (using unmodified MWNT) was nearly unchanged compared to pure epoxy. The aminoepoxy composite was electrically conductive (average resistivity 2k-Ohms/cm) but dependent on percolation paths caused by partial aggregation of the aligned nanotubes in the composite. The structure and properties of composites using VA-MWNTs infiltrated with other polymers will be discussed.

Fig 1.

TOP VIEW

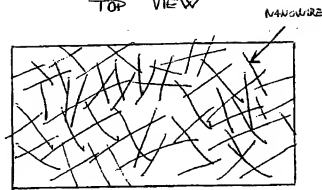


Fig 2.

SIDE VIEW

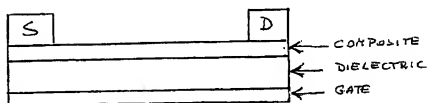
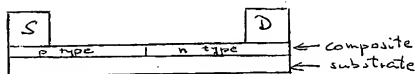


Fig 3.



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